BSI Standards Publication

Iron ores — Determination of various elements by X-ray fluorescence spectrometry

Part 2: Single element calibration procedure

Documents
Document Sharing Hub

Full document details: PD ISO/TS 9516-2:2024

National foreword

This Published Document is the UK implementation of ISO/TS 9516-2:2024.

The UK participation in its preparation was entrusted to Technical Committee W/-, Consumer Products and Services Sector Policy and Strategy Committee.

A list of organizations represented on this committee can be obtained on request to its committee manager.

Contractual and legal considerations

This publication has been prepared in good faith, however no representation, warranty, assurance or undertaking (express or implied) is or will be made, and no responsibility or liability is or will be accepted by BSI in relation to the adequacy, accuracy, completeness or reasonableness of this publication. All and any such responsibility and liability is expressly disclaimed to the full extent permitted by the law.

This publication is provided as is, and is to be used at the recipient's own risk.

The recipient is advised to consider seeking professional guidance with respect to its use of this publication.

This publication is not intended to constitute a contract. Users are responsible for its correct application.

This publication is not to be regarded as a British Standard.

© The British Standards Institution 2024
Published by BSI Standards Limited 2024

ISBN 978 0 580 91731 8

ICS 73.060.10

Compliance with a Published Document cannot confer immunity from legal obligations.

This Published Document was published under the authority of the Standards Policy and Strategy Committee on 31 May 2024.

Amendments/corrigenda issued since publication

Date	Text affected

Documents
Document Sharing Hub

Full document details: PD ISO/TS 9516-2:2024

Technical Specification

ISO/TS 9516-2

First edition
2024-05

Iron ores — Determination of various elements by X-ray fluorescence spectrometry —

Part 2: Single element calibration procedure

Reference number
ISO/TS 9516-2:2024(en)

© ISO 2024

Documents
Document Sharing Hub

Full document details: PD ISO/TS 9516-2:2024

COPYRIGHT PROTECTED DOCUMENT

© ISO 2024

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Documents
Document Sharing Hub
Full document details: PD ISO/TS 9516-2:2024

Contents

Page

Foreword ... **v**
1 Scope ... **1**
2 Normative references ... **1**
3 Terms and definitions ... **1**
4 Principle ... **2**
5 Reagents and materials ... **2**
6 Apparatus ... **4**
7 Sampling and samples ... **5**
7.1 Laboratory sample ... 5
7.2 Preparation of test samples ... 5
7.2.1 General ... 5
7.2.2 Method specified in ISO 2596 ... 5
7.2.3 Method specified in ISO 7764 ... 6
8 Procedure ... **6**
8.1 General ... 6
8.2 Number of determinations ... 7
8.3 Check analysis and blank test ... 7
8.4 Preparation of discs ... 7
8.4.1 Weighing ... 7
8.4.2 Mixing ... 8
8.4.3 Fusion ... 8
8.4.4 Casting ... 8
8.4.5 Visual inspection ... 8
8.4.6 Disc storage ... 8
8.4.7 Cleaning of platinum ware ... 9
8.4.8 Test discs ... 9
8.5 Measurement ... 9
8.5.1 General ... 9
8.5.2 Effect of errors or omissions ... 10
8.5.3 Analytical lines ... 11
8.5.4 XRF generator settings ... 11
8.5.5 Crystals ... 14
8.5.6 Line overlaps ... 14
8.5.7 Collimators ... 14
8.5.8 Pulse height settings ... 14
8.5.9 Counting time ... 14
8.5.10 Simultaneous instruments ... 15
8.5.11 Sample holders ... 15
8.5.12 Measurement sequence ... 16
8.6 Drift correction ... 16
8.6.1 Preparation of drift correction monitor discs ... 16
8.6.2 Drift correction using monitor disc ... 17
9 Calculation of results ... **19**
9.1 General ... 19
9.2 Calibration equation ... 20
9.3 Calculation of alpha coefficient ... 23
9.4 Correction for sample, flux and oxidizer mass ... 24
9.4.1 General ... 24
9.4.2 Correction of flux/sample mass ratio ... 24
9.4.3 Correction of remaining oxidizer/sample mass ratio ... 24
9.4.4 Mass ratio correction constant in calibration Equation 1 ... 25
9.5 Calculation of calibration ... 25

© ISO 2024 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TS 9516-2:2024

9.5.1 Preparation of calibration discs ... 25
9.5.2 Calculation of concentrations of calibration discs ... 28
9.5.3 Measurement of calibration discs ... 29
9.5.4 Calculation of calibration coefficients ... 29
9.6 Calculation of concentrations ... 33
9.6.1 Calculation of flux/sample and remaining oxidizer/sample mass ratios ... 33
9.6.2 Calculation of initial concentration ... 34
9.6.3 Calculation of concentrations ... 34
9.6.4 Conversion from oxide to element concentrations ... 35

10 General treatment of results ... 35
10.1 Background equivalent concentration (BEC) ... 35
10.2 Determination of analytical result ... 36
10.3 Check for trueness ... 36
10.4 Calculation of the final result ... 37
10.5 Oxide factors ... 38

11 Test report ... 38

Annex A (normative) **Preparation of Flux A and Flux D ... 39**

Annex B (normative) **Preparation of Flux B and Flux C ... 41**

Annex C (normative) **Standard deviation of specimen preparation ... 42**

Annex D (normative) **Spectrometer precision tests ... 44**

Annex E (informative) **Theoretical derivation of correction term in calibration ... 48**

Annex F (informative) **Calculation of correction coefficients using the FP method (fundamental parameter method) ... 53**

Annex G (informative) **Calculation of calibration coefficients and correction alphas by the least square method ... 56**

Annex H (informative) **Calculation of counting time ... 60**

Annex I (informative) **Air cooling block for fused discs ... 63**

Annex J (normative) **Flowchart for acceptance of results ... 64**

Bibliography ... 65

© ISO 2024 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TS 9516-2:2024

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

A list of all parts in the ISO 9516 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

© ISO 2024 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TS 9516-2:2024

Documents
Document Sharing Hub

Iron ores — Determination of various elements by X-ray fluorescence spectrometry —

Part 2:
Single element calibration procedure

WARNING — This document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This document sets out a wavelength dispersive X-ray fluorescence procedure for the determination of various elements in iron ores. The method is applicable to iron ores regardless of mineralogical type.

2 Normative references

The following documents are referred to in the text in such a way that some of all of their content constitutes requirements of this document. For dated reference, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 2596, *Iron ores — Determination of hygroscopic moisture in analytical samples — Gravimetric, Karl Fischer and mass-loss methods*

ISO 3082, *Iron ores — Sampling and sample preparation procedures*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 7764, *Iron ores — Preparation of predried test samples for chemical analysis*

ISO 8655-2, *Piston-operated volumetric apparatus — Part 2: Pipettes*

ISO 11323, *Iron ore and direct reduced iron — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11323 apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses.

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

© ISO 2024 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TS 9516-2:2024

4 Principle

The glass discs for X-ray fluorescence measurement are prepared by incorporating the test portion of the iron ore sample, via fusion, into a borate glass disc using a casting procedure. By using a fused glass disc, particle size effects are eliminated. Sodium nitrate may be added to the flux to ensure complete oxidation of all components, particularly iron and sulfur. Any of four methods for glass disc preparation may be used: three use lithium borate as flux; the other uses sodium borate.

X-ray fluorescence measurements are based on the "line only" principle. If desired, backgrounds can be measured to obtain net line intensities. This method is applicable to data from simultaneous and sequential X-ray fluorescence spectrometers.

This method relies on measuring all components of the sample, other than volatiles. If some components are not measured, errors will result in the measured components (see 8.5.2).

Results are obtained after matrix corrections for inter-element effects.

5 Reagents and materials

During analysis, only reagents of recognized high purity, and only grade 2 water as specified in ISO 3696 shall be used.

Where reagents have been ignited, they shall be stored during cooling in a desiccator and weighed as soon as possible.

5.1 Silicon dioxide, (SiO_2), nominally 99,999 % SiO_2.

The silicon dioxide shall contain less than 3 µg/g of each of the other elements listed in Table 1. It shall be heated to 1 000 °C in a platinum crucible for a minimum of 2 h and cooled in a desiccator.

5.2 Aluminium oxide, (Al_2O_3), analytical reagent grade, α form.

If the α form is used, it shall be heated to 1 000 °C in a platinum crucible for a minimum of 2 h. If the aluminium oxide is not the α form, it shall be converted to the α form by heating to 1 250 °C in a platinum crucible for a minimum of 2 h. It shall be cooled in a desiccator and weighed as soon as it is cooled.

5.3 Iron(III) oxide, (Fe_2O_3), purity of 99,995 % or more Fe_2O_3.

The iron(III) oxide shall contain less than 3 µg/g of each of the other elements listed in Table 1. It shall be heated at 1 000 °C in a platinum crucible for a minimum of 1 h and cooled in a desiccator.

5.4 Titanium dioxide, (TiO_2).

Analytical grade titanium dioxide shall be heated at 1 000 °C in a platinum crucible for a minimum of 1 h and cooled in a desiccator.

Phosphorus is a common impurity in TiO_2 and a reagent low in phosphorus shall be selected. The selected reagent shall be checked, as even nominally high-purity reagents can be significantly contaminated, e.g. a supposed 99,99 % TiO_2 grade reagent has been found to contain about 0,5 % P_2O_5.

5.5 Potassium dihydrogen orthophosphate solution, (KH_2PO_4).

Potassium dihydrogen orthophosphate KH_2PO_4 of purity 99,0 % or more shall be dried at 105 °C for 1 h and cooled in a desiccator. 3,481 g of the dried potassium dihydrogen orthophosphate shall be dissolved in 100 ml water in a volumetric flask. One ml of this solution contains 7,92 mg phosphorous and 10,0 mg potassium.

© ISO 2024 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TS 9516-2:2024

5.6 Calcium carbonate, ($CaCO_3$).

Analytical grade calcium carbonate shall be dried at 105 °C for 1 h and cooled in a desiccator.

5.7 Manganese (II) sulfate hydrate solution, ($MnSO_4 \cdot 5H_2O$).

8,777 (= 5,497 + 5 × 0,656) g of manganese sulfate hydrate $MnSO_4 \cdot 5H_2O$ of purity 99 % or more shall be dissolved in 100 ml water in a volumetric flask. Manganese sulfate monohydrate ($MnSO_4 \cdot H_2O$) may be used as an alternative reagent of purity 99 % or more and 6,153 (= 5,497 + 0,656) g of the reagent shall be dissolved in 100 ml water in a volumetric flask. One millilitre of the solution contains manganese 20,0 mg and sulfur 11,67 mg.

5.8 Magnesium nitrate hexahydrate, [$Mg(NO_3)_2 \cdot 6H_2O$], purity of 99,0 % or more.

5.9 1 000 mg/l element standards for V, Cr, Co, Ni, Cu, Zn, As, Pb and Ba.

Single element standard water solutions with 1 000 mg/l concentration and 0,5 to 6 % of nitric acid shall be used. It is recommended to use commercially available standards. When standard solutions are prepared in the laboratory, 1,000 g of pure metal (99,99 % or more) shall be dissolved in diluted nitric acid. Water shall be added to this solution such that the prepared standard solution is 1 000 ml.

For the standard water solution for Cr, a solution of potassium dichromate solution ($K_2Cr_2O_7$) with nitric acid may be used.

5.10 Sodium nitrate, ($NaNO_3$).

Analytical grade sodium nitrate shall be dried at 105 °C for 1 h and cooled in a desiccator.

5.11 Ammonium iodide, (NH_4I).

Laboratory reagent grade ammonium iodide does not need to be dried but shall be stored in a desiccator.

5.12 Desiccant.

The desiccant shall be freshly regenerated self-indicating silica gel.

5.13 Flux.

5.13.1 General

One of fluxes from Flux A, Flux B, Flux C or Flux D, as described in 5.13.2, 5.13.3 and 5.13.4, shall be used. The levels of contamination in the flux shall be checked (see 10.1). Because levels of contamination can vary from batch to batch, the same batch of flux shall be used for all discs (iron ore, blank and calibration) involved in the batch of determinations.

5.13.2 Flux A and Flux D

Flux A and Flux D shall be prepared by fusion of a mixture of anhydrous lithium tetraborate ($Li_2B_4O_7$) and anhydrous lithium metaborate ($LiBO_2$) using the procedure specified in Annex A. Flux shall be dried at 500 °C for a minimum of 4 h and stored in a desiccator.

5.13.3 Flux B

Flux B shall be prepared using sodium tetraborate using the procedure specified in Annex B. Flux shall be dried at 500 °C for a minimum of 4 h and stored in a desiccator.

© ISO 2024 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TS 9516-2:2024

5.13.4 Flux C

Flux C shall be prepared using lithium tetraborate using the procedure specified in Annex B. Flux shall be dried at 500 °C for a minimum of 4 h and stored in a desiccator.

NOTE If this flux is used, sulfur will not be reported.

6 Apparatus

The sample may be fused with the flux in a crucible and then poured into a separate mould or, if an appropriately shaped crucible is used, the fusion may be carried out and the glass allowed to cool in the same crucible. Both methods will produce glass discs of the same quality.

A conventional electric furnace, high-frequency furnace, or a gas burner may be used for heating. There are disc-making machines commercially available, and these may be used to fuse and cast the discs.

A platinum lid may be used to cover the crucible if fusing in a furnace, but not if fusing over a flame, as this enhances sulfur loss.

Where a high-frequency furnace or a gas burner is used for heating, a check shall be made to determine if sulfur is lost during disc preparation. A mixture that contains 90 % Fe_2O_3 and 10 % $CaSO_4$ shall be prepared and used to prepare replicate discs using normal fusion times and times of twice and thrice normal. The intensity of SKα from the discs should not vary by more than 2 % relative.

Single-volume pipettes, one-mark volumetric flasks and piston pipettes to be used shall comply with specifications of ISO 648, ISO 1042 and ISO 8655-2, respectively.

6.1 Analytical balance, capable of weighing to the nearest 0,1 mg.

6.2 Crucible and mould.

The crucible and mould shall be made from a non-wetting platinum alloy. Either platinum/gold or platinum/gold/rhodium alloys are suitable.

If more than one crucible or more than one mould is used for casting, these crucibles or moulds shall all be used in the specimen preparation test in Annex C. It is essential to use all of the crucibles or moulds in the test described in Annex C, as casting vessels can become distorted with use, giving the analytical surface a curvature that will result in error.

Sometimes, even undistorted crucibles or moulds give curvatures unique to the particular crucible or mould.

6.2.1 Crucible

Where the crucible is used for fusion only, it shall have sufficient capacity to hold the flux and sample required for fusion. Where the crucible is used as a mould as well as for fusion, it shall have a flat bottom, to enable production discs with minimum curvature.

6.2.2 Mould

Because the bottom of the disc is the analytical surface, the inside bottom surface of the mould shall be flat and shall be polished regularly with approximately 3 µm diamond paste to ensure that the glass disc releases easily from the mould. To prevent deformation through repeated heating and cooling, the base shall be greater than 2 mm thick. The mould shall have a flat bottom, to enable production of discs with minimum curvature.

6.3 Electric furnace, capable of maintaining a temperature of at least 1 050 °C.

The furnace shall be capable of maintaining higher temperatures where it is to be used for converting Al_2O_3 to the α form (1 250 °C), or for preparing Flux A (1 100 °C).

© ISO 2024 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TS 9516-2:2024

The furnace may be of a conventional type with heating elements, or may be a high-frequency furnace. The furnace shall be cleaned regularly to prevent contamination of the samples.

6.4 Gas-oxygen burner.

Where fusions are made over a gas-oxygen flame, provision shall be made for oxygen enhancement of the flame to minimize sulfur loss and crucible contamination. The temperature of the melt shall be in the range 1 000 °C to 1 050 °C. The temperature shall be checked using an optical pyrometer while the crucible contains several grams of flux. Alternatively, if an optical pyrometer is not available, about 3 g of potassium sulfate (m.p. 1 069 °C) shall be put in an empty crucible and the flame adjusted so that it all just melts in the open crucible. A gas burner may be used for heating the mould, and it shall be adjusted so that the mould is a bright red heat (approximately 950 °C). A Meker burner shall not be used, as loss of sulfur and the uptake of iron from the glass into the platinum ware can result.

6.5 Desiccator.

6.6 Spatulas, non-magnetic, for weighing of the test portion and for mixing.

6.7 X-ray fluorescence spectrometer, of any wavelength dispersive, vacuum (or helium) path, simultaneous or sequential type, X-ray fluorescence spectrometer, provided that the instrument has been checked.

Performance checks shall be carried out in accordance with the precision tests set out in Annex D, accumulating at least 2×10^7 counts for each measurement.

6.8 Ultrasonic bath, optional. It may be used to aid cleaning of the platinum ware.

6.9 Cooling device.

It is recommended that the mould and glass be cooled using an air jet. Commercial disc-making machines use this method. A drawing of a suitable device is given in Annex I.

Whatever the method of cooling, it is vital that samples be treated identically, as the curvature of the analytical surface of the disc depends on the rate of cooling.

7 Sampling and samples

7.1 Laboratory sample

For analysis, laboratory samples of –100 µm particle size which has been taken and prepared in accordance with ISO 3082 shall be used. In the case of ores having significant contents of combined water or oxidizable compounds, samples of particle size of –160 µm shall be used.

7.2 Preparation of test samples

7.2.1 General

Depending on the ore type, air-equilibrate test samples shall be prepared in accordance with either ISO 2596 in 7.2.2 or ISO 7764 in 7.2.3.

7.2.2 Method specified in ISO 2596

The method is applicable to all types of ores.

The laboratory samples shall be thoroughly mixed, and in multiple increments, a test sample shall be extracted in such a manner that it is representative of the entire content in the container. The test sample shall be brought into equilibrium with the laboratory atmosphere in accordance with ISO 2596.

© ISO 2024 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TS 9516-2:2024

7.2.3 Method specified in ISO 7764

The method is not applicable to the following types of ores:

a) processed ores containing metallic iron;

b) natural or processed ores in which the sulfur content is higher than 0,2 %;

c) natural or processed ores in which the content of combined water is higher than 2,5 %.

The laboratory samples shall be thoroughly mixed and, multiple increments shall be taken, a test sample shall be extracted in such a manner that it is representative of the whole contents of the container.

8 Procedure

8.1 General

The concentration range covered for each of the component elements is given in Table 1.

Table 1 — Range of application of the method

Component element	Concentration range for referee purposes %	Concentration range for analysis %
Fe	31 to 72	31 to 72
Si	0,16 to 11,8	0,16 to 11,8
Ca	0,011 to 13,7	0,011 to 13,7
Mn	0,016 to 2,0	0,016 to 2,0
Al	0,036 to 4,2	0,036 to 4,2
Ti	0,013 to 4,5	0,013 to 4,5
Mg	0,012 to 1,7	0,012 to 1,7
P	0,001 3 to 0,6	0,001 3 to 0,6
S	0,011 to 0,76	0,011 to 0,76
K	0,008 to 0,46	0,008 to 0,46
V	0,002 to 0,32	0,002 to 0,32
Cr	—	0,006 to 0,067
Co	—	0,006 to 0,023
Ni	0,008 to 0,038	0,008 to 0,038
Cu	0,007 to 0,17	0,007 to 0,17
Zn	0,005 to 0,36	0,005 to 0,36
As	—	0,003 to 0,11
Pb	—	0,001 to 0,23
Ba	—	0,011 to 0,74

When the influence of absorption or spectral overlap by trace heavy elements to reporting elements is small enough and can be ignored, those trace heavy elements can be omitted from measuring elements.

The applicable ranges for referee and non-referee methods are also to be determined based on results of international trials.

The operator shall have demonstrated the ability to consistently make discs with high precision. This ability shall be verified by carrying out the procedure given in Annex C.

The operator shall periodically test all moulds according to Annex C, because their shape can become distorted with repeated use.

© ISO 2024 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TS 9516-2:2024

In preparing discs, great care shall be taken to avoid contamination and, in particular, the crucible in which the fusion is carried out shall be thoroughly cleaned prior to use (see 8.4.7).

8.2 Number of determinations

Carry out two analyses independently for duplicate fused discs prepared on different days in accordance with Annex J for each test sample (see 7.2).

8.3 Check analysis and blank test

In each run, one analysis of a certified reference material of the same type of ore shall be carried out in parallel with the analysis of the ore sample(s) under the same conditions. A test sample of the certified reference material shall be prepared in the manner appropriate to the type of ore involved.

When analysis is carried out on several samples of the same ore type at the same time, the analytical result of one certified reference material may be used.

When a new reagent as for flux from a different bottle is used, it is recommended to measure a blank sample to check the impurities and contamination due to sample preparation, before the analysis is carried out on ore sample(s). The blank disc shall be prepared with 100 % Fe_2O_3.

8.4 Preparation of discs

8.4.1 Weighing

Table 2 shows the components used in making the glass discs. Provided that the proportions are kept approximate to those given in Table 2, the masses can be varied to suit mould diameter. If a disc diameter used differs from those given in Table 2, masses should be adjusted to be approximately proportional to the area of the glass disc.

Table 2 — Masses of specimen components

Component	Typical masses g	Mass g	
		Disc diameter	
		32 mm	40 mm
Flux	6,80	3,52 to 6,40	5,50 to 10,00
$NaNO_3$	0,40 or 0	0,21 to 0,37 or 0	0,33 to 0,58 or 0
Sample	0,66	0,35 to 0,60	0,55 to 0,94

The specified masses may be weighed as “catch” weights, recording the mass weighed to the nearest 0,001 g for the flux and sodium nitrate portions, and to the nearest 0,000 1 g for the test and calibration portions. If masses used are higher than recommended, crystallization and segregation with consequent cracking are likely to occur as the glass cools.

If desired, ammonium iodide (5.11) can be used as a releasing agent. If added at this stage, no more than 0,01 g shall be added. Alternatively, a smaller amount may be added prior to casting (see 8.4.4). Exceeding the recommended amount of releasing agent of ammonium iodide can introduce error in the titanium result due to the overlap. $IL\beta_2$ interferes with TiKα.

Because the components are hygroscopic, they shall be weighed as soon as possible after reaching room temperature following heating and without any undue delay between each weighing. Weighing may be made directly into the crucible to be used in the fusion, or into a clean glass vial. Because of static effects, glass vials are preferable to plastic. If a vial is used, care shall be taken to ensure complete transfer of the contents into the fusion crucible.

© ISO 2024 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TS 9516-2:2024

8.4.2 Mixing

Thoroughly mix the components in the crucible using a spatula or similar implement, taking care that no material is lost. The mixing implement used should be free of sharp or pointed edges, in order to ensure that the interior of the crucible is not damaged by scratching. Brush any fine material adhering to the mixing implement back into the crucible. Gently tap the bottom of the crucible on the bench top to ensure that any material adhering to the crucible wall, above the general level of the mixed components, is reincorporated into the bulk of the mix.

It is imperative that the crucible be tapped gently on the bench top, as too severe an impact will result in the loss of some of the finer material and possible deformation of the crucible. Care shall be taken to mix the components thoroughly to aid reproducibility of disc preparation.

8.4.3 Fusion

For samples containing sulfur as sulfide, the fusion mixture is to be preoxidised by heating to 700 °C for 10 min prior to fusion. Place the crucible in the electric furnace (6.3) or on the gas-oxygen burner (6.4) at a temperature of 1 000 °C to 1 050 °C and maintain this temperature for 10 min. At least once during this period, after the sample is dissolved, briefly swirl the mixture. While swirling, incorporate into the melt any material that can be adhering to the sides of the crucible.

If a furnace is used for heating, it can be necessary to remove the crucible from the furnace for the purpose of swirling. When the furnace is opened, the temperature can drop. The specified temperature shall be regained before the time period starts.

8.4.4 Casting

If ammonium iodide was not added as a release agent earlier, it may be added to the melt just prior to casting.

In this case, no more than 0,002 g shall be added. Casting is then carried out by one of the following methods.

a) Casting in the crucible.

   If the glass is to be cast in the crucible, remove the crucible from the furnace, place on a suitable cooling device (6.9) and allow the glass to solidify.

b) Casting in a separate mould.

   If the glass is to be cast in a separate mould, the mould shall be pre-heated over a gas flame to red heat (900 °C to 1 050 °C). While the mould is still hot, pour the melt into the mould from the crucible. Remove the mould from the heat source and place it on the cooling device (6.9) and allow the glass to solidify.

NOTE Failure to ensure that the mould is scrupulously clean prior to casting will result in discs sticking to the mould and possibly cracking.

8.4.5 Visual inspection

Prior to storage, discs shall be inspected visually, paying particular attention to the analytical surface. The discs shall not contain undissolved material, and shall be whole and free from crystallization, cracks and bubbles. Defective discs shall be re-fused in the crucible, or discarded and substitute discs prepared.

8.4.6 Disc storage

As soon as possible (while the glass is still warm), transfer the discs to a desiccator so that absorption of moisture and the possibility of contamination are minimized. When not being measured, discs shall be stored in a clean desiccator.

To avoid contamination of the analytical surface, the specimen shall be handled by its edges and the surface shall not be touched by hand or treated in any way. Specifically, it shall not be washed with water or other solvents, ground or polished.

© ISO 2024 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TS 9516-2:2024

If paper labels are used on the backs of discs, great care should be taken to ensure that the labels do not contact the analytical surfaces of other discs. Paper labels are clay coated and readily cause contamination by silicon and aluminium. For the same reason, paper envelopes should not be used to store the discs.

8.4.7 Cleaning of platinum ware

Although the crucible and mould are fabricated from an alloy that is not wetted by the glass, in order to ensure absolute precision, they shall be cleaned between each fusion. Immersion in hot hydrochloric, citric or acetic acid (approximately 2 M), for about 1 h is usually sufficient, but they should be inspected to ensure that all residual glass has been removed.

A rapid method of cleaning is to put the crucible or mould into a beaker containing the acid. Place the beaker in a small ultrasonic bath for about 1 min or until all residual glass is removed, then rinse the mould in distilled water and dry before using.

An alternative method of cleaning is to fuse several grams of flux in the crucible, moving the melt around to clean the entire inner surface. The molten flux is then poured from the crucible. If a droplet adheres to the crucible, this can easily be flaked off when the crucible is cold.

If new platinum ware is used without pre-conditioning by the cleaning described above, then the Fe results will be unreliable until conditioning occurs. New platinum ware can also release contaminants without conditioning.

8.4.8 Test discs

One disc from each test sample shall be prepared. At least one certified reference material, of the same type as the ore used in the test discs, shall be prepared. Prior to fusing test discs, crucibles should be thoroughly clean, particularly if the same crucibles were used to prepare the calibration discs, some of which are high in trace elements.

8.5 Measurement

8.5.1 General

The analytical lines to be used and suggested operating conditions are given in Table 3. Other instrument parameters (collimators and detectors) shall be selected according to the particular element.

Table 3 — Suggested analytical lines, crystals and operating conditions

Component element	Line (see 8.5.3)	Voltage (see 8.5.4) kV	Crystal [a] (see 8.5.5)	Specific line overlaps (see 8.5.6)
Fe	Kα Kβ	40 to 80 40 to 80	LiF(200) or LiF(220) LiF(200) or LiF(220)	— —
Si	Kα	25 to 50	PE	—
Ca	Kα	25 to 50	LiF(200) or PE or Ge(111)	—
Mn	Kα	50 to 80	LiF(200)	CrKβ
Al	Kα	25 to 50	PE	BaLα(3), CrKβ(4)
Ti	Kα	40 to 80	LiF(200)	BaLα
Mg	Kα	25 to 50	TlAP or multi-layer	—
P	Kα	25 to 50	Ge(111) or PE	—
S	Kα	25 to 50	Ge(111) or PE	CoKα(3), PbMα
K	Kα	25 to 50	LiF(200)	—
V	Kα	50 to 80	LiF(200)	TiKβ, BaLβ

[a] The first crystal listed is preferred.

© ISO 2024 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TS 9516-2:2024

Table 3 *(continued)*

Component element	Line (see 8.5.3)	Voltage (see 8.5.4) kV	Crystal [a] (see 8.5.5)	Specific line overlaps (see 8.5.6)
Cr	Kα	50 to 80	LiF(200)	VKβ
Co	Kα	50 to 80	LiF(200)	FeKβ
Ni	Kα	50 to 80	LiF(200)	CoKβ
Cu	Kα	50 to 80	LiF(200)	—
Zn	Kα	50 to 80	LiF(200)	—
As	Kα Kβ	50 to 80 50 to 80	LiF(200) LiF(200)	PbLα —
Pb	Lβ1 Mα	50 to 80 25 to 50	LiF(200) Ge(111) or PE	— FeKα(3)
Ba	Lα Lβ1	40 to 80 40 to 80	LiF(200) LiF(200)	TiKα TiKβ, VKα

[a] The first crystal listed is preferred.

8.5.2 Effect of errors or omissions

There are various circumstances where all 19 elements will possibly not be determined. If a simultaneous instrument is used, there will possibly not be analytical channels for all elements. In plant control, it is probably not necessary to do all the minor elements. Where the source material is constant and known, it is probably unnecessary to do the minor elements on all samples.

When converting intensities to concentrations, the intensities are multiplied by the term (the matrix factor) of absorption and enhancement effects and this factor receives contributions from all components of the sample, so if one or more components are not determined then all other components will be in error. The method then relies on the measurement of all components of the sample.

Table 4 shows the error, as a relative percentage, for each analyte where there is a 1 % error in a component.

The error can be not estimating a component if it has a significant concentration. The calculations have been made for a typical iron ore.

Table 4 can be used to estimate at what level a minor element can be omitted from the analysis without exceeding a predetermined error.

Where small errors are involved in a component, the resulting errors to other components are proportional to the initial error, and if more than one component is in error, or omitted, the errors are additive.

The errors shown in Table 4 have been calculated on the basis of matrix errors only. Overlap errors can give rise to additional errors. In such cases, the error to a component is an absolute concentration, i.e. the error is not proportional to the concentration of the analyte. Overlap errors are more important for minor elements. No attempt has been made to quantify these errors as they are dependent on instrument parameters. See Table 3 for possible line overlaps.

Iron is the element required with high precision, and if 0,1 % Fe_2O_3 is regarded as the maximum error that can be tolerated then, using Table 4, it can be seen that the omission of measuring 250 ppm BaO will give such an error. From Table 4, it can be seen that an error of 1 % in BaO gives a relative error of 2,97 % in Fe_2O_3, so that if the Fe_2O_3 content of the ore is 90 % the resulting error due to 0,025 % BaO is calculated as Formula (1):

$$0{,}025 \times 2{,}97 \times 90/100 = 0{,}067\ \% \qquad (1)$$

© ISO 2024 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TS 9516-2:2024

8.5.3 Analytical lines

Line-only positions are measured. It is not necessary to measure background intensities, but if desired they can be measured and net intensities recorded.

8.5.4 XRF generator settings

The voltage, in kilovolts, is not critical and normally with a simultaneous instrument will be set in the range 40 kV to 60 kV. Where using a sequential instrument, it can be advantageous to use a low voltage (40 kV) for the lighter elements and a higher voltage for the heavier elements. It should be noted that certain spectrometers cannot be run at the highest voltage specified in Table 3, and it is not necessary to do so. If tube operating conditions are changed during analysis, this can result in slight instability in the spectrometer output. Since Fe content is required to be determined with very high precision, Fe shall be measured in a separate run with constant tube conditions if conditions vary for the other elements.

When XRF generators are powered up, it is common for the instrument to drift for some time, typically 30 min to 60 min. Therefore, prior to measurement, the generator should be powered up and left to stabilize.

All measurements shall be made under vacuum, using a detector (proportional or scintillation counter) appropriate to the wavelength being measured, and using specimen rotation if available. A Cr, Cr/Au, Sc, Sc/Mo, Sc/W or Rh target X-ray tube shall be used. It is recommended that pulse height selection be used, particularly in the case where low concentrations are being determined. Where count rates are very high (e.g. FeKα or CaKα), either wide pulse height settings or no upper level shall be used.

In special circumstances (e.g. determining Cr or Mn using a Cr type tube), primary beam filters may be used. A filter will be required to achieve low backgrounds for the determination of Mn and Cr using a Cr target X-ray tube.

© ISO 2024 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TS 9516-2:2024

Table 4 — Error, in relative %, resulting from a 1 % error in a component for flux

Case	Compound																					
a)	Inter-fered	Affected																				
		Fe_2O_3	SiO_2	CaO	Mn_3O_4	Al_2O_3	TiO_2	MgO	P_2O_5	SO_3	K_2O	SnO_2	V_2O_5	Cr_2O_3	Co_3O_4	NiO	CuO	ZnO	As_2O_3	PbO	BaO	Cl
	SiO_2	0,37	---	0,31	0,28	0,10	0,31	0,09	0,27	0,27	0,30	0,30	0,32	0,32	0,26	0,14	0,14	0,14	0,13	0,13	0,31	0,28
	CaO	1,25	0,12	---	0,91	0,12	0,98	0,12	0,12	0,12	0,18	0,27	1,01	1,03	0,87	0,49	0,49	0,49	0,48	0,47	0,98	0,13
	Mn_3O_4	0,56	0,19	0,18	---	0,19	0,19	0,18	0,19	0,19	0,17	0,17	0,19	0,38	1,36	0,78	0,79	0,79	0,79	0,78	0,18	0,19
	Al_2O_3	0,33	0,24	0,27	0,24	---	0,28	0,08	0,24	0,25	0,27	0,27	0,28	0,28	0,22	0,13	0,12	0,12	0,12	0,11	0,28	0,25
	TiO_2	1,24	0,14	0,19	0,90	0,14	---	0,13	0,14	0,14	0,17	0,17	0,37	1,01	0,86	0,49	0,49	0,49	0,49	0,49	0,34	0,14
	MgO	0,30	0,24	0,26	0,22	0,23	0,26	---	0,24	0,24	0,25	0,25	0,26	0,26	0,20	0,11	0,11	0,11	0,10	0,10	0,26	0,24
	P_2O_5	0,41	0,11	0,34	0,30	0,10	0,34	0,09	---	0,29	0,33	0,33	0,34	0,34	0,28	0,16	0,16	0,15	0,15	0,14	0,34	0,30
	SO_3	0,45	0,12	0,37	0,33	0,11	0,37	0,10	0,13	---	0,35	0,36	0,37	0,37	0,31	0,17	0,17	0,17	0,16	0,16	0,37	0,32
	K_2O	1,24	0,11	0,99	0,90	0,11	0,96	0,11	0,11	0,11	---	0,27	0,99	1,01	0,86	0,49	0,49	0,49	0,48	0,47	0,95	0,12
	SnO_2	1,96	0,23	0,54	1,42	0,22	1,50	0,21	0,24	0,26	0,44	---	1,56	1,59	1,37	0,78	0,78	0,78	0,78	0,75	1,50	0,28
	V_2O_5	1,29	0,15	0,18	0,94	0,14	0,31	0,14	0,15	0,15	0,17	0,17	---	0,40	0,89	0,51	0,51	0,51	0,50	0,50	0,32	0,15
	Cr_2O_3	1,74	0,17	0,20	0,39	0,17	0,25	0,17	0,17	0,17	0,19	0,20	0,42	---	1,21	0,69	0,69	0,69	0,69	0,69	0,25	0,17
	Co_3O_4	0,59	0,23	0,21	0,12	0,22	0,21	0,22	0,23	0,23	0,22	0,21	0,21	0,21	---	0,30	0,99	1,00	1,01	1,02	0,21	0,23
	NiO	0,34	0,27	0,28	0,24	0,26	0,28	0,26	0,27	0,28	0,28	0,29	0,31	0,32	0,49	---	0,36	1,17	1,21	1,23	0,30	0,28
	CuO	0,33	0,29	0,31	0,25	0,28	0,30	0,27	0,29	0,30	0,30	0,31	0,32	0,33	0,23	0,29	---	0,38	1,28	1,30	0,32	0,30
	ZnO	0,35	0,32	0,34	0,27	0,31	0,33	0,30	0,32	0,33	0,34	0,35	0,35	0,36	0,23	0,05	0,33	---	1,43	1,46	0,35	0,34
	As_2O_3	0,43	0,38	0,44	0,35	0,33	0,43	0,10	0,39	0,40	0,43	0,43	0,45	0,44	0,28	0,08	0,05	0,03	---	1,58	0,44	0,41
	PbO	1,98	0,29	1,37	1,45	0,24	1,57	0,20	0,34	0,42	1,23	1,26	1,63	1,66	1,38	0,74	0,73	0,73	0,97	---	1,57	0,92
	BaO	2,97	0,35	0,62	1,92	0,32	0,84	0,28	0,37	0,39	0,56	0,58	0,97	1,74	2,07	1,19	1,19	1,20	1,21	1,22	---	0,42
	Cl	1,09	0,10	0,84	0,80	0,08	0,86	0,08	0,11	0,13	0,80	0,81	0,88	0,90	0,75	0,43	0,43	0,42	0,41	0,40	0,86	---
	Na_2O	0,26	0,21	0,22	0,20	0,21	0,23	0,20	0,21	0,21	0,22	0,22	0,23	0,23	0,18	0,10	0,10	0,10	0,09	0,09	0,23	0,21

NOTE 1 Case a) is the estimated errors calculated for Flux C and 10,0 of flux/sample mass ratio. The estimated errors of Flux A and D are almost identical to them.

NOTE 2 Case b) is the estimated errors calculated for Flux B and 10,0 of flux/sample mass ratio.

© ISO 2024 – All rights reserved

Full document details: PD ISO/TS 9516-2:2024

AVA Documents
Document Sharing Hub

Table 4 *(continued)*

Case	Compound																				
b)	Inter-fered	Affected																			
		Fe_2O_3	SiO_2	CaO	Mn_3O_4	Al_2O_3	TiO_2	MgO	P_2O_5	SO_3	K_2O	SnO_2	V_2O_5	Cr_2O_3	Co_3O_4	NiO	CuO	ZnO	As_2O_3	PbO	BaO	Cl
	SiO_2	0,23	---	0,21	0,19	0,07	0,21	0,06	0,19	0,19	0,20	0,20	0,21	0,21	0,18	0,12	0,12	0,11	0,11	0,10	0,21	0,19
	CaO	0,77	0,08	---	0,63	0,08	0,65	0,08	0,08	0,08	0,12	0,18	0,67	0,68	0,60	0,40	0,39	0,39	0,38	0,35	0,65	0,09
	Mn_3O_4	0,35	0,13	0,12	---	0,13	0,12	0,13	0,13	0,13	0,12	0,11	0,12	0,25	0,95	0,63	0,63	0,63	0,62	0,59	0,11	0,13
	Al_2O_3	0,20	0,17	0,19	0,17	---	0,19	0,06	0,17	0,17	0,18	0,18	0,19	0,19	0,16	0,10	0,10	0,10	0,10	0,09	0,19	0,18
	TiO_2	0,77	0,10	0,12	0,62	0,10	---	0,10	0,09	0,10	0,11	0,11	0,23	0,67	0,60	0,40	0,40	0,40	0,39	0,37	0,21	0,10
	MgO	0,19	0,17	0,18	0,15	0,16	0,17	---	0,17	0,17	0,17	0,17	0,17	0,17	0,14	0,09	0,09	0,09	0,09	0,09	0,17	0,17
	P_2O_5	0,25	0,08	0,23	0,21	0,07	0,22	0,07	---	0,21	0,22	0,22	0,23	0,23	0,20	0,13	0,13	0,12	0,12	0,11	0,22	0,21
	SO_3	0,28	0,08	0,25	0,23	0,08	0,25	0,07	0,09	---	0,24	0,24	0,25	0,25	0,21	0,14	0,14	0,14	0,13	0,12	0,25	0,23
	K_2O	0,77	0,08	0,67	0,62	0,08	0,64	0,08	0,08	0,08	---	0,18	0,66	0,67	0,60	0,40	0,39	0,39	0,38	0,36	0,64	0,08
	SnO_2	1,22	0,16	0,36	0,98	0,15	1,00	0,15	0,17	0,18	0,29	---	1,03	1,06	0,96	0,63	0,63	0,63	0,62	0,58	1,00	0,19
	V_2O_5	0,80	0,10	0,12	0,64	0,10	0,20	0,10	0,10	0,10	0,11	0,11	---	0,25	0,62	0,41	0,41	0,41	0,39	0,37	0,21	0,10
	Cr_2O_3	1,07	0,12	0,13	0,27	0,12	0,15	0,12	0,12	0,12	0,12	0,12	0,27	---	0,84	0,56	0,56	0,55	0,54	0,51	0,15	0,12
	Co_3O_4	0,37	0,16	0,14	0,09	0,16	0,13	0,16	0,16	0,16	0,14	0,14	0,13	0,13	---	0,24	0,80	0,80	0,79	0,75	0,13	0,16
	NiO	0,19	0,19	0,19	0,16	0,19	0,18	0,18	0,19	0,19	0,19	0,19	0,19	0,19	0,34	---	0,29	0,94	0,94	0,91	0,19	0,20
	CuO	0,19	0,20	0,20	0,16	0,20	0,19	0,20	0,21	0,21	0,20	0,21	0,20	0,20	0,14	0,24	---	0,30	1,00	0,96	0,20	0,21
	ZnO	0,20	0,23	0,23	0,17	0,22	0,21	0,21	0,23	0,23	0,23	0,23	0,22	0,22	0,15	0,04	0,27	---	1,11	1,08	0,22	0,24
	As_2O_3	0,26	0,27	0,29	0,23	0,23	0,28	0,07	0,27	0,28	0,29	0,29	0,29	0,28	0,19	0,07	0,05	0,03	---	1,17	0,29	0,29
	PbO	1,22	0,20	0,93	0,99	0,17	1,04	0,14	0,24	0,29	0,84	0,85	1,07	1,09	0,96	0,60	0,59	0,59	0,77	---	1,04	0,64
	BaO	1,84	0,24	0,41	1,32	0,23	0,54	0,20	0,26	0,27	0,37	0,38	0,63	1,14	1,44	0,96	0,96	0,96	0,95	0,91	---	0,29
	Cl	0,68	0,07	0,57	0,55	0,06	0,57	0,05	0,08	0,09	0,55	0,55	0,59	0,60	0,53	0,35	0,34	0,34	0,32	0,30	0,57	---
	Na_2O	0,17	0,15	0,15	0,14	0,15	0,15	0,15	0,15	0,15	0,15	0,15	0,15	0,15	0,13	0,08	0,08	0,80	0,08	0,08	0,15	0,15
	Na_2O	0,17	0,15	0,15	0,14	0,15	0,15	0,15	0,15	0,15	0,15	0,15	0,15	0,15	0,13	0,08	0,08	0,80	0,08	0,08	0,15	0,15

NOTE 1 Case a) is the estimated errors calculated for Flux C and 10,0 of flux/sample mass ratio. The estimated errors of Flux A and D are almost identical to them.

NOTE 2 Case b) is the estimated errors calculated for Flux B and 10,0 of flux/sample mass ratio.

© ISO 2024 – All rights reserved

Full document details: PD ISO/TS 9516-2:2024

AVA Documents Document Sharing Hub

8.5.5 Crystals

The crystals listed in Table 3 are those preferred for the measurements, particularly for sequential-type instruments. Other crystals could, however, be used if these are not available. In the case of PKα, the Ge(111) crystal is recommended because it does not give second-order wavelengths. If, however, this crystal is unavailable, and a PE crystal is used, pulse height selection shall be used and the settings very carefully selected, so that the possibility of interference from the second-order wavelength of CaKβ is minimized.

8.5.6 Line overlaps

Relevant specific line overlaps are shown in Table 3. Note that certain well-known overlaps are not shown since the overlapping element is low in the materials under consideration.

8.5.7 Collimators

Coarse or fine collimators can be used, but where there is the likelihood of line interference, a fine collimator should be used (e.g. in the determination of manganese using a chromium target X-ray tube, and the determination of phosphorus using a PE crystal). In general, the fine collimator will give lower overlap interferences and BECs.

Where crystal fluorescence contributes significantly to BEC, it is also advisable to use a fine collimator, e.g. magnesium determinations using TlAP (thallium acid phthalate) crystal and Rh tube.

8.5.8 Pulse height settings

Where there is interference from a second order line, narrow pulse height settings should be used to minimize interference. Also, where crystal fluorescence contributes to background, it may be possible to reduce the BEC by using a narrower than normal pulse height analyser window setting.

8.5.9 Counting time

The standard deviation of X-ray intensity can be estimated by determining the square root of total counts. This generally applies to relatively low count rates (about 300 kc/s or lower). At higher count rates the counting statistical error is worse than calculated. Follow the recommendation of the instrument manufacturer. The calculation procedure to obtain the counting time for an element to measure with a required precision by counting statistics is described below.

The dedicated calibration curve for the determination of counting time shall be obtained for each element. The calibration is obtained from the concentrations and X-ray intensities of two calibration samples for each element.

Iron	Fe_2O_3 100 %
Other than Iron	Calibration standard sample of the element and blank (Fe_2O_3 100 %)

For iron, background intensity is ignored to estimate counting time.

The counting time of iron can be calculated by Formula (2):

$$T = \{100 \times [1 + \alpha_{\mathrm{FeFe}} (s_i + 100)]\}^2 / \left(s_i^2 \times I_{\mathrm{Fe}}\right) \quad (2)$$

© ISO 2024 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TS 9516-2:2024

where

T is the measuring time (s);

s_i is the specified precision of analyte i (%);

I_{Fe} is the intensity of Fe of Fe_2O_3 100 % disc;

α_{FeFe} is the alpha coefficient of Fe_2O_3 for Fe_2O_3 analysis calculated using fundamental parameter method.

The number of 100 in Formula (2) is 100 % of Fe_2O_3 concentration in Fe_2O_3 100 % disc.

For the elements other than iron, the calibration curve without correction is defined as Formula (3).

$$C_i = A_i I_i - B_i \quad (3)$$

where

C_i is the concentration of analyte i (%);

I_i is the X-ray intensity of analysing element i (c/s);

A_i, B_i are the calibration coefficients of analyte i.

The counting time to get the specified precision is obtained by Formula (4).

$$T = A_i (C_i + B_i) / s_i^2 \quad (4)$$

where

T is the measuring time (s);

C_i is the concentration of analyte i (%);

s_i is the specified precision of analyte i (%).

Formula (4) represents the calculation of counting time without background subtraction. It is necessary to consider the statistical variation of background intensity when background subtraction is performed for an element. At high count rates, the time T required shall be lengthened. Follow the manufacturer's recommendations of the instrument.

Annex H describes examples of counting time calculation.

8.5.10 Simultaneous instruments

Where using simultaneous-type instruments, the manufacturer will supply crystals to determine each element.

These possibly will or will not correspond to those listed in Table 3.

Likewise, there is no selection of collimators for simultaneous instruments. The slit size will be predetermined by the manufacturer.

8.5.11 Sample holders

Sample holders for disc presentation shall be matched in accordance with the reproducibility test specified in Annex D.

© ISO 2024 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TS 9516-2:2024